SUBJECT INDEX

ab initio method 66–8, 73
ACCORDION experiment 39, 48, 49
Acids and their derivatives, TMS tagging 314–16
Additivity rule 301
Adipose tissue 529–30
Albumins 515–16
Alcohols, TMS tagging 316–23
Alkynes 209–53
  chemical shifts 214–26
  coupling constants 235–46
  solid state NMR 247–8
Alkynyl carbon atoms 214–15
AMBER program 133
AMKX system 24, 33
Angle χ^1 127
Anisotropic chemical shielding (CSA) 570–4
Anisotropy 556–8
ARMA process 263–4, 285
Assignment strategies 311–13
  utilizing isotopic labeling 441–9
Assignment techniques 299–313
Atomic inversions 166–71, 189–90
Autoregressive-moving average process. *See* ARMA process
Average Hamiltonian theory 5–8, 15
Average root mean square distances (rmsd) 133–4

Backbone chain motion 587
Bandshape analysis 147–8
Basic pancreatic trypsin inhibitor (BPTI) 113
Bimetallic Pt/Rh clusters, bonding of CO to 419–20
BINEPTR 304, 313
Biopolymers 423–52
Bis-phenol A polycarbonate. *See* BPAPC
Bloch decay line shapes 570–4
Blood, outer sphere relaxation 544–6
Bond rotations 172–6, 190–2
BPAPC 570, 591, 593, 594, 597–9
Brownian rotational diffusion 584–5

^{13}C assignments 113–15
^{13}C chemical shifts 214–15
^{13}C labeled proteins 107–9
^{13}C NMR spectroscopy 211–13
  of solids 247
  synthetic polymer systems 349–400
Calibration of cross peak intensities between different mixing times 118
Calibration of cross relaxation rates with distances 118–20
C≡C triple bond 215
Chelate complexes 538–9
Chemical shift 351–3
Chemical shift anisotropy (CSA) 570
Chemical shift dispersion 135
Chemical shift scaling 15, 17
Chemisorbed molecules, mobility of 419
Chemisorbed species 402–5
CO, bonding to Pt/Rh bimetallic clusters 419–20
Coal products, TMS tagging 340–4
Cole–Cole expression 495, 496, 502
COMATOSE program 98–9, 121, 161
Complete relaxation matrix analysis. *See* CORMA
Concertina effect 14–17
Conducting polymers, secondary structures in the solid state 380–6
Conduction electrons 419
CORMA 84–7, 121
  distance determinations 90–9
Correlated spectroscopy. *See* COSY spectra
Correlation leakage 275
COSS spectra 29–32
COSY spectra 58, 102, 441
  basic pulse sequence 22–4
  relaxation effects 34–6
  scaling modifications 25–34
Coupling constants 123–8
  alkynes 235–46
  heteronuclear 124–5
  homonuclear 125–7
  measurement of 44–8
Cross-linked proteins 515–19
Cross-relaxation 522–4
  ^{14}N–proton 517
  data 161
  proton–proton 503–10, 516–17
  vs hydroxynamic interactions in tissue 530–1
CSA interaction 570–5
CSA line shape studies 570–5
CSA line shapes, overlapping 574
CSA powder 581
CSA spin echo line shapes 574–5
CYCLOPS 305

DANTE pulse sequence 148
Denatured proteins 515–19
Density operator 8–11, 27
Deoxyribonucleic acid (DNA) 3
DEPT 292, 295–307
Deuterium powder 579–81
Deuterium quadrupolar solid echo line shapes 575–7
Deuterium spin alignment 577–8
Deuterons, pure water 493–4

Diamagnetic systems 492–532
Diamagnetic tissue 489–90
Dihedral angle constraints 132–3
Dihydroxy alcohols, TMS tagging 320–3
Diluent effects on polymer motions 598–600
Diluent motions in polymer–diluent systems 601
Dilute limit 494–512
Dipolar rotational spin echo ^{13}C NMR 578–9
Discrete Fourier transform 256–61
Discrete large angle jumps 583
DISGEO program 76, 133
DISMAN program 76, 133
Distance calculations 87–90
Distribution of correlation times 585–7
DNMR spectra 142, 144, 147, 188, 189, 194, 196
D_2O solution 105
DQF-COSY 24, 58
Dynamic MAS NMR spectroscopy 582
Dynamic NMR. *See* DNMR

Electronic parameters 554–6
EMBED method 133
Energy diagram for two-spin system 117
Energy evaluation methods 73
Exchange and molecular symmetry 144–7
Exchange rates, measurement of 48–50
Exchanges 186–9, 198–208
EXSY, recent developments 164–5
EXSYM program 146
Eye lens protein calf γ_{II}-crystallin 514

Ferritin, outer sphere relaxation 543
FID 255, 257–9, 261, 263, 264, 267–9, 272–4, 285
Field-cycling relaxometry 487–567, 561
Field gradient, rapid motion in 418–19
Fingerprint method 66
Fluxional rearrangements 177–86
Functional group analysis 291

Gram–Schmidt orthogonalization procedure (MGS) 265

^1H assignments 102–13
  first sequential assignments 102–5
  homonuclear sequential assignment strategies 105–7
  using heteronuclear editing techniques with ^{15}N or ^{13}C labeled proteins 107–9
^1H NMR spectroscopy 213
^1H–^{13}C–^{15}N–^1H triple resonance techniques 110–11
Hall–Helfand correlation function 587
Hamiltonian 4–8, 12–13, 18
Hammett-type correlations 301
Hard-sphere exo-anomeric (HSEA) calculations 75
Hemoglobin 512–14
Heteronuclear correlation 109–13
Heteronuclear three-dimensional spectroscopy 112
Heteronuclear two-dimensional chemical shift correlation 309–11
Heteronucleus–heteronucleus correlation experiments 439–41
Hexoses, TMS tagging 334–7
Highly hindered hydroxy groups, silylation of 314
HOHAHA (homonuclear Hartman–Hahn correlation) 61, 433
Homogeneous globular protein solutions 494–515
Homogeneous tissue 519–24
Homonuclear three-dimensional spectroscopy 112
Homonuclear Hartman–Hahn spectroscopy (HOHAHA) 61, 433
Homonuclear sequential assignment strategies 105–7
Householder's method (QRD) 265
Hydrodynamic interactions in tissue 530–1
Hydrogen bond constraints 133
Hydrogen bonds 128

INADEQUATE 307, 440
INEPT 292, 295–307
Inflection field 496
Inhomogeneous tissue 524–30
Inner sphere relaxation
  in dilute solutions 546–58
  of vesicles 558–60
Inner sphere waters 553–4
Insoluble polymers, secondary structures in the solid state 388
Internal molecular motions 161
Intramolecular mutual exchange 144
Intraprotein diffusion 506–9
Intraresidue H^N–H^α connectivities 105
Inverse gated decoupling (IGD) 295
Inverse power law dispersion 522–4
Isolated spin pair approximation (ISPA) 88–9
Isotopic labeling 423–52
  assignment of resonances of proteins using 441–9
  assignment strategies utilizing 441–9
  early applications 425–6
  preparation methods 426–9
Isotopic substitution 302
Iterative relaxation matrix approach (IRMA) 91

J-scaling 12–14

Knight shift 411–13

Lanthanide-induced shifts 302–3
Least Squares (LSQ) method 255, 259–61, 265, 266, 268, 272, 273, 277, 278, 280
Lignins, TMS tagging 337–40
Line shapes 411–17
Line width measurement in CSPMAS ^{13}C NMR 582
Linear prediction (LP) method 255, 262–86
Local minimum problem 72
Long-range correlations 43–4
Long-range hydrodynamic interaction 511–12

Macroscopic viscosity 512
Magic angle spinning NMR 416–17
Magnetic resonance imaging (MRI) 489–91
Magnetite particulates 539–40
Magnetization relaxation 506–9
Magnetization transfer experiments 148–50
Main-chain-directed (MCD) assignment strategy 107
Matrix analysis of rates for DIscerning geometRy in solution (MARDIGRAS) 91–9
Maximum Entropy Method (MEM) 255
Microscopic viscosity 512
MINDOR (modified INDOR) 303
Molecular dynamics
  one-dimensional NMR spectroscopy 147–50
  simulations 76, 78
  two-dimensional NMR spectroscopy 150–64
Molecular mechanics methods 73–5
Molecular orbitals 419
Molecular stereodynamics 141–208
Molecular symmetry 144–7
Monohydroxy alcohols, TMS tagging 316–20
Motional narrowing 413–16, 492–3
Multi-dimensional NMR 424–5
Multiple quantum coherences (MQC) 39–40
Multiple quantum transitions (MQT) 1, 40–1
Myelinated white matter 524–9

^{15}N assignments 113–15
$^{15}N/^{13}C$ double labeling 109
^{15}N labeled proteins 107–9, 124–5
^{15}N–$^{1}H\alpha$ long-range correlation 112
^{14}N–proton cross-relaxation 517
Nitroxide radicals 537–8
NMR tagging 290–4
NMR time scales 142–4
NMRD profiles 519–24
NOEs 4, 22, 132, 154
  and mobility 122
  averaging effects vs correlation time effects 122
  for structure determinations 115–22
  identification of large number of 115
  one-dimensional measurements 68
  relations between cross peaks, cross relaxation rates, distances, spectral density functions and distance ranges 115–17
NOESY spectra 22, 61–2, 69, 102, 441
  back calculation with complete relaxation matrix approach 121
  basic pulse sequence 36–9
  integrating cross peaks 120
  recent developments 164–5
  scaling modifications 38–9
  zero-quantum contributions in cross peaks of *J*-coupled nuclei 120
Non-selective polarization transfer 295–307
Nuclear magnetic resonance. *See* NMR
Nuclear Overhauser effect difference spectroscopy (NOEDS) 57
Nuclear Overhauser effect spectroscopy. *See* NOESY spectra
Nuclear Overhauser effects. *See* NOEs
Nuclear Overhauser enhancement (NOE) 36
Nuclear spin relaxation 210

Off-resonance heteronuclear decoupling 11–14
Oligosaccharide–ligand interactions 79
Oligosaccharides
  binding and interaction 78–9
  combined NMR/theoretical methods 76–7
  conformations 55–81
  determination of primary sequence 65–8
  determination of three-dimensional structure 68–77
  heteronuclear methods 64
  importance of 3D structure to function 78
  molecular motion 77–8
  one-dimensional NMR spectroscopy 56–8
  resonance assignments in 56–65
  stereospecific assignments 64–5
  strong coupling 63–4
  structural studies 56
  three-dimensional NMR spectroscopy 62–3
  TMS tagging 330–4
  two-dimensional NMR spectroscopy 58–62
One-dimensional NMR spectroscopy
  molecular dynamics 147–50
  oligosaccharides 56-8
  recent applications 165–203
One-dimensional NOE measurements 68
One-dimensional scaling experiments 11–17
Outer sphere relaxation
  blood 544–6
  dilute solutions of spherical dipoles 533–43
  ferritin 543

Paraffinic-chain containing polymers 380
Paraffins
  crystal structures 373–4
  crystalline and noncrystalline components 370–3

folded structures 374–7
side branches and end groups 379–80
Paramagnetic centers 135
Paramagnetic complexes, binding to exterior of vesicles 561
Paramagnetic spheres 536–7
Paramagnetic systems 532–61
Paramagnetic tissue 490
Pattern recognition 68
Pentoses, TMS tagging 330–4
Peptide bonds 446–7
Petroleum products, TMS tagging 340–4
pH range 135
Phenols, TMS tagging 323–30
Polyethers
secondary structures in the solid state 388
stereochemical configuration and conformation 358–60
Polyethylene
crystal structures 373–4
crystalline and noncrystalline components 370–3
folded structures 374–7
oriented 377–9
side branches and end groups 379–80
Polyhydroxy alcohols, TMS tagging 320–3
Polymer–diluent systems, diluent motions in 601
Polyolefins
secondary structures in the solid state 386–7
stereochemical configuration and conformation 355–7
Polypropylene, secondary structures in the solid state 386–7
Polyvinyl chloride, stereochemical configuration and conformation 358–60
Product operator descriptions 11
Product operator evolution 9–10
Product operator formalism 8–11
Product operator properties 11
Protein degradation 135
Protein mobility 135
Protein model building algorithms 133
Protein–protein interactions 512–15
Protein size effect 134
Protein structures 101–39
criteria for judging quality of 133–4
data sets for structure calculations 132–3
identification of secondary structures 129–131
limitations for NMR studies 134–5
procedures for characterizing secondary structures 129–30
tertiary structures 131–3
Proteins 425
assignments of resonances using 441–9
Proton detection spectroscopy 429–34
experimental considerations 434–6
three-dimensional 437–9
two-dimensional 436–7
Proton–proton cross-relaxation 503–10, 516–17
Proton–proton two-dimensional NMR experiments 441
Protons
early phenomenology for 494–502
pure water 492
Pure water 492–4
deuterons 493–4
protons 492

Quantitative distances from initial slopes 118–20
Quantitative spectral analysis of NMR data 255–88

R factors 134
Random rotational jumps 583
Redfield limit 492
Regioirregularity, vinyl polymers 360
Relaxation effects 34–6
Relaxation matrix analysis of two-dimensional NMR data 83–100
Relaxation times
distribution of 417
measurement of 581–2
Relaxometry 489
RELAY 58–60, 441
Residual violations of constraints 133–4
Resonance assignments in oligosaccharides 56–65
Restrained molecular dynamics calculations (RMD) 133
Restricted rotational diffusion 585
RF pulses 10
Ring conformational changes 192
ROESY 69, 163, 164, 441

Saccharides, TMS tagging 330–7
Scalar coupled systems 160–1
Scaling
concept of 1
NMR parameters 4–5
spin–spin coupling constants 18
SCUBA 105–7
Secondary structures, impact of identification on structure calculations 130–1
SECSY spectra 34, 44, 58
SEDOR 408–11, 420
Segmental motion 587–93
Selective ^{1}H–^{29}Si decoupling 303
Selective ^{29}Si–^{13}C decoupling 307
Selective ^{29}Si–^{1}H decoupling 303
Selective heteronuclear INADEQUATE 308–9
Selective INEPT 305
Semi-empirical methods 73
Semi-selective ^{1}H–^{29}Si decoupling 303
Shielding order 301
^{29}Si NMR spectroscopy 289–348

Sidechain assignment strategies 447–9
Silylation methods 313–14
Simultaneous jump and oscillation 585
Single correlation time 585–7
Slow beat measurements 406–8
Solid state ^{13}C NMR spectroscopy 350
Solid state MAS-NMR spectroscopy 482
Solid state NMR spectroscopy
  local motions in polymers 569–603
  of alkynes 246–7
Solution ^{13}C NMR spectroscopy 350
Solvent effects 75
Spin coupling constant measurements 70
Spin-decoupling difference spectroscopy (SDDS) 57
Spin-echo correlated spectroscopy (SECSY) 34, 44, 58
Spin-echo double resonance. *See* SEDOR
Spin-lattice relaxation 417–19
Spin–spin coupling 353–4
SPINEPTR 304, 313
Stereospecific assignments 127–8
Stokes' Law 496
Substitutent group rotation 593–8
Surface phenomena 401–21
Synthetic fuels, TMS tagging 340–4
Synthetic polymer systems
  ^{13}C NMR spectroscopy 349–400
  local motions in 569–603
  primary structures 354–70
  secondary structures in solution 354–70
  secondary structures in the solid state 370–95
Synthetic polypeptides, secondary structures in the solid state 389–95

Tagging technique 290
τ_m values 118
Three-dimensional NMR spectroscopy, oligosaccharides 62–3
TMS derivatives, measurement of ^{29}Si spectra 294–9
TMS tagging 289–348
  acids and their derivatives 314–16
  advantages of 293–4
  alcohols 316–23
  coal products 340–4
  dihydroxy alcohols 320–3
  hexoses 334–7
  lignins 337–40
  monohydroxy alcohols 316–20
  NMR properties of 294
  oligosaccharides 330–4
  pentoses 330–4
  petroleum products 340–4
  phenols 323–30
  polyhydroxy alcohols 320–3
  results and applications 314–44
  saccharides 330–7
  synthetic fuels 340–4
TOCSY spectra 34, 433, 441
Transition metal surfaces 404–5
Trimethylsilyl (TMS) tags. *See* TMS tagging
Trimethylsilylation 313–14
Triple-quantum filtered COSY (TQF-COSY) 60–1
Two-dimensional correlated spectroscopy 21–2
Two-dimensional EXSY spectra 150–7
Two-dimensional *J*-resolved spectroscopy
  heteronuclear case 19–21
  homonuclear case 18–21
Two-dimensional line shapes 579–81
Two-dimensional multiple quantum spectroscopy
  basic pulse sequence 39–41
  scaling modifications 41–2
Two-dimensional NMR spectroscopy 3–4, 272–85, 351
  basic experimental scheme 3–4
  categories of techniques 4
  detection periods 17
  molecular dynamics 150–64
  oligosaccharides 58–62
  recent applications 165–203
  relaxation matrix analysis data 83–100
Two-dimensional scaling experiments 17–42
  applications 43–50
Two-site model 510–11

Universal NMRD profile 521

Vanadium
  basic properties 453–6
  biological applications 480–2
  closed-shell 462–82
  couplings 482–3
  open-shell 457–61
Vanadium halo complexes 465–7
Vanadium nitrato complexes 478–80
Vanadium nitrogen ligands 480
Vanadium oxo complexes 468–75
Vanadium peroxo complexes 478–80
Vanadium selenido-complexes 464–5
Vanadium sulphido-complexes 464–5
Vanadium-51 NMR 453–85
Vanadyl anhydrides 475–8
Vanadyl esters 475–8
Variable target function method 133
Vesicles
  exterior bound paramagnetic ions 561
  inner sphere relaxation 558–60
  with entrapped paramagnetic agents 558–61
Vinyl polymers, stereochemical configuration and conformation 354–60
Virtual conformations 78
Volatile compounds, silylation of 314

WAHUHA scheme 578

Z-COSY spectra 34